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# REMOVAL OF Cr(VI) FROM SIMULATED ELECTROPLATING WASTEWATER BY MAGNETITE NANOPARTICLES

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## Abstract

In this study, the efficiency of magnetic nanoparticles for removal of hexavalent chromium from simulated electroplating wastewater was evaluated. The nanoparticles were prepared using the sol-gel method and were characterized by X-ray diffraction (XRD), X-ray fluorescence (XRF), a scanning electron microscopy energy dispersive X-ray analyzer (SEM-Edx), a particle sizer and a vibrating sample magnetometer (VSM). The results showed that synthesized nanoparticles were in the size range of 40-300 nm, had purity of about 90 percent, and had magnetization of 36.5 electromagnetic unit per gram (emu/g). In conditions including pH 2, Cr (VI) concentration of 10 mg/L, nanomagnetite concentration of 1 g/L, a shaking speed of 250 rpm and a 20 minute retention time, 82% of Cr(VI) was removed. Competition from common coexisting ions such as $Na^+$, $Ni^{2+}$, $Cu^{2+}$, $NO_3^-$, $SO_4^{2-}$, and $Cl^-$ was negligible. The adsorption data was well fitted by the Freundlich isotherm. We conclude that magnetite nanoparticles have considerable potential for removal of Cr(VI) from electroplating wastewaters.

*Key words:* adsorption, chromium, industrial wastewater, magnetite nanoparticles

*Received:*

## 1. Introduction

Nanotechnology is considered to be one of the most important advancements in science and technology in the past decade. It is related to the manipulation of materials and systems at the nanometer scale. At this length scale, materials exhibit unique properties that can be used for novel applications like magnetization (Uheida et al., 2006; Pop et al., 2009). Metallic elements have been excessively released into the environment due to rapid industrialization and have created a major global concern (Wan Ngah and Hanafiah, 2008; Bulai et al., 2009). Hexavalent chromium, Cr(VI), is a hard oxidant and a group A carcinogen. The industrial sources of Cr(VI) primarily include alloy and steel manufacturing, metal finishing, electroplating, leather tanning, and pigments synthesis and dyeing industries (Wang and Lo, 2009; Bulgariu et al., 2008). The effluents from these industries contain Cr(III) and Cr(VI) at concentrations ranging from tenths to hundreds of mg/L (Thacker et al., 2006).

Contact with chromium can result in severe health problems ranging from simple skin irritation to lung carcinoma (Shen et al., 2009), therefore it should not be present in the leather fabrics. A variety of methods have been developed for removal of chromium compounds from industrial wastewater. Hexavalent chromium usually exists in wastewater as oxyanions such as chromate ($CrO_4^{2-}$) and dichromate ($Cr_2O_7^{2-}$) and does not precipitate easily using conventional precipitation methods. Currently,

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chemical redox followed by precipitation is the most widely used technique for Cr(VI) removal. This is achieved by reduction of hexavalent chromium to a less toxic trivalent state by applying a reducing agent, such as sodium metabisulfite, sulfur dioxide gas, or ferrous sulfate, and subsequent precipitation of the trivalent chromium. This approach has three direct disadvantages: high waste treatment equipment costs, high consumption of reagents, and high volume of sludge generated. This method also has two indirect disadvantages: it is a potential hazard to the environment due to landfill leachate, and using this method akems difficult to recover metals for re-use (Hu et al., 2005a). The use of magnetic nanoparticles for separation and pre-concentration in analytical chemistry is a new methodology that is faster, simpler, and more precise than those used traditionally. The greatest advantage of this new method is that desired materials are separated from a solution by a simple and compact process, producing less secondary waste. Other advantages are large active surface area for a given mass of particles, and the ability to process a solution that contains suspended solids (Khajeh and Khajeh, 2009). Magnetic carriers for metal ion removal from dilute solutions such as mining drain waters, natural pollutions, and hazardous wastes are effective tools allowing remote and safe operations (Hristov and Fachikov, 2007).

Nowadays, there are a few articles on the effect of magnetically assisted Gas–Solid fluidization on removal of heavy metal and pollutants from water and wastewater (Hristov, 2008). Iron oxides in various forms have been used for the removal of radioactive and metallic elements from water and wastewater solutions (Borai et al., 2007). In addition, the removal and recovery of Cr (VI) has been achieved with synthesized magnetite ($Fe_3O_4$) nanoparticles, and Cr(VI) adsorption capacity of nanoscale magnetite compared favorably with other adsorbents, such as activated carbon and clay (Hu et al., 2005a). The objective of the present work was to investigate the role and the effectiveness of synthesized magnetic nanoparticles ($Fe_3O_4$) for the removal of Cr(VI) from simulated electroplating wastewater. In this work, the magnetic nanoparticles were first synthesized by the sol-gel method. Then, different variables were experimentally manipulated, including the amount of Cr(VI) loaded on the nanoparticles, the pH, the adsorption time, the shaking rate, the concentration of magnetic nanoparticles, the adsorption isotherm equation and the competition among coexisting ions.

## 2. Experimental

### *2.1. Chemicals and reagents*

The chemicals used in this study were primarily reagent or higher grades and were used as received without further treatment. The chemicals used were as follows: $K_2Cr_2O_7$ (>99.9%, Merck), $FeCl_3$ - $6H_2O$ $FeCl_2$, $4H_2O$ (>99%, Merck), 25% ammonia solution (Merck), NaOH (Merck). All chemical solutions were prepared with deaerated deionized water (DDW) by deoxygenating 18 MΩcm of deionized water with 99.9% nitrogen for 2 h.

### *2.2. Synthesis and characterizations of the adsorbent*

To prepare the nanoscale magnetite, it was important to conduct a reaction (Eq. 1) by combining a molar ratio of Fe(II)/Fe(III) = 0.5 in a basic solution.

$$2FeCl_3 + FeCl_2 + 8NH_3 + 4H_2O \rightarrow Fe_3O_4 + 8NH_4Cl \quad (1)$$

In the laboratory, magnetite nanoparticles were prepared by developing the sol–gel method. First, 200mL of purified deoxygenated water (resistivity of 18MΩ) was bubbled by nitrogen gas for 30 min. Then 5.2 g $FeCl_3$ and 2.0 g $FeCl_2$ were dissolved in the mixture with mechanical stirring. Under the protection of nitrogen gas, 1.5 mol/L $NH_3$ (aq) solution was added dropwise into the mixture under vigorous stirring. Initially a brown precipitate formed, followed by a black precipitate. When pH reached 8.0, the stirrer was turned off and the magnetite settled gradually. The black precipitate was isolated by an external magnetic field, and the supernatant was decanted. To obtain pure and neutral products, synthesized materials were washed with ultrapure water several times. Finally, the wet nanogel was freeze-dried (Hu et al., 2004).

A scanning electron microscope (SEM) equipped with an energy dispersive X-ray analyzer (EDX) (Seron, AIS-2100, South Korea) was used in this study. The composition of the materials were identified by X-ray fluorescence (XRF) (Bruker pioneer-S4, Germany) and X-ray diffraction (XRD) (Bruker, D8ADVANCE, Germany), this instrument was equipped with copper anode generating (Cu -Kα) radiation (λ= 1.5406 Å).

The particles sizes and the zeta potentials of magnetite nanoparticles were measured with a particle sizer and a zeta potential meter (Malvern, England). Magnetization measurements were performed at room temperature using a vibrating sample magnetometer (VSM) in the Development Center of Kashan University.

### *2.3. Magnetic separations*

Magnetic separations were performed with a magnetic field column separator including two magnets and a plexiglass column, 90 cm height, 5 cm diameter, and 1.78 L volume. The glass column was packed with 50 g stainless-steel wool (approximately 80μm wire diameter). The sample was passed through the column, so the nanoparticles were retained in the column as the solution passed through (Mayo et al., 2007). Nanoparticles were collected for further tests.

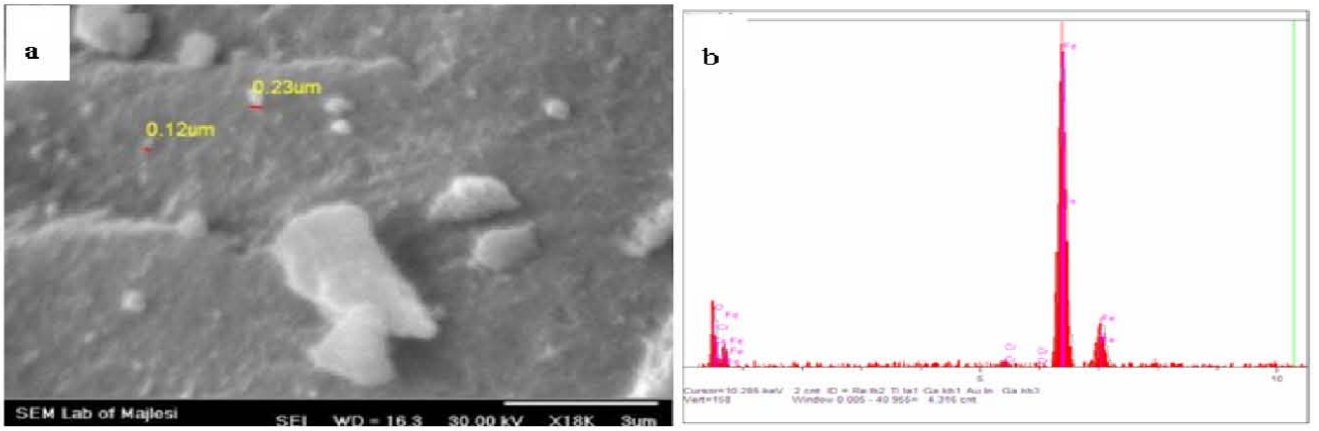


**Fig. 1.** (a) SEM image of magnetic nanoparticles,
(b) The SEM-EDX image spectra of magnetite nanoparticles after chromium adsorption

The average magnetic field intensity on the axis separator tube approximately 0.13 Tesla (T) obtained. By flux meter, F-50 WALKER (USA).

### *2.4. Batch tests*

Batch adsorption studies were performed by mixing and agitating 0.1 g magnetite nanoparticles with 20 mL $K_2Cr_2O_7$ solution of varying concentration in a 100 mL erlenmeyer flask containing 0.1g of sorbent. Standard acid (0.1M $HNO_3$) and base (0.1M NaOH) solutions were used for pH adjustment. All the adsorption experiments were carried out at room temperature (22.5°C) and a pH of 2, unless stated otherwise. Adsorption isotherm studies were conducted by varying the initial Cr (VI) concentration from 10 to 200 mg/L at different levels of pH (2, 5, 7, and 9). To explore the competitive effects of various coexisting ions (e.g., $Na^+$, $Cu^{2+}$, $Ni^{2+}$, $NO_3^-$, $SO_4^{2-}$ and $Cl^-$) on the removal of Cr(VI), 20 mL of 50 mg/L Cr(VI) solution containing combinations of these ions in two concentrations (50 and 100 mg/L) and 0.1 g of magnetite nanoparticles were shaken at pH 2. After the adsorption reached equilibrium, the adsorbent was separated via an external magnetic field and the supernatant was collected for metal concentration measurements. All experiments were performed in duplicate and the averaged values were taken. The concentrations of chromium were measured by an inductively coupled plasma optical emission spectrometer (ICP-OES, Ultima-2, France) (Hu et al., 2005b).

## 3. Results and discussion

### *3.1. Characterization of adsorbent*

The SEM image of the particles (Fig. 1.a) revealed that the synthesized nanoparticles in this study were multi-dispersed in the size range of 40-300 nm, with two specified particles of 120 and 230 nm (0.12 and 0.23 μm).

The SEM-EDX image spectra of the nanoparticles after adsorption are shown in Fig. 1.b. The characteristics and purity of the magnetite nanoparticles were evident because XRD peaks of the nanocrystallite matched well with standard $Fe_3O_4$ and other crystalline phases were not detected (Fig. 2). The results of X-ray fluorescence (XRF) of our nanoparticles are shown in Table 1. The purity of produced nanoparticles was approximately 90 percent.

The paramagnetic properties of the magnetic particles were verified with the magnetization curve measured by VSM (Fig. 3). The saturation moment of the synthesized particles, from the hysteresis loop measured by VSM, was found to be 36.5 emu/g. The zeta potential of magnetite nanoparticles was approximately -6.85mv (ZETA-potential meter, model, MALVERN).

**Table 1.** XRF analysis of nanoparticles produced in this study

| *Compound* | *Concentration (%W/W)* |
|---|---|
| $Fe_3O_4$ | 89.61 |
| Cl | 4.09 |
| CuO | 0.048 |
| ZnO | 0.024 |
| LOI* | 7.02 |
| **Total** | **100.79** |

*Loss on Ignition

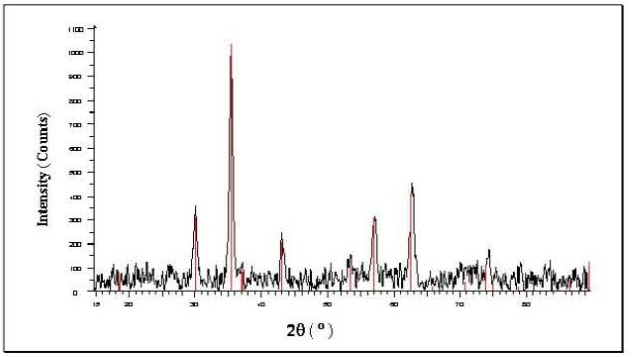


**Fig. 2.** XRD patterns of magnetite particles

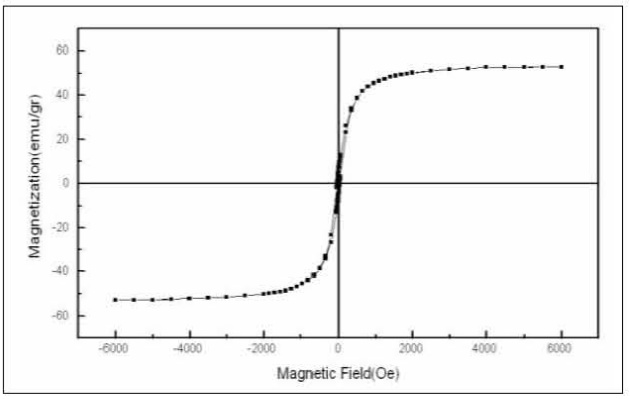


**Fig. 3.** Magnetization curves of nanoparticles synthesized

### *3.2. Effect of pH on chromium removal efficiency*

The pH of the solution clearly affected removal efficiency. The optimum pH was 2. As can be seen in Fig. 4, the average removal efficiency of Cr(VI) at pH levels of 2, 5, 7 and 9 was 82%, 77%, 59% and 39%, respectively. The rate of chromium removal is reduced as the pH in the solution increases. Studies by Qian also showed reduced chromium removal efficiency with increasing pH (Qian et al., 2008).

The reaction occurred in a short period of time and at a wide range of pH levels Approximately 90% of the Cr(VI) was removed during the first minute of the reaction. Only a small part of the additional removal occurred during the following 14 minutes of contact. The rapid adsorption of Cr(VI) by magnetite nanoparticles is perhaps attributed to the external surface adsorption, which is different from the micro-porous adsorption process. Due to the fact that nearly all of the adsorption sites of magnetite nanoparticles exist in the exterior of the adsorbent, compared to the porous adsorbent, it is easy for the adsorbate to access these active sites. This results in a rapid approach to equilibrium. With increasing pH in the reaction mixture, uptake of Cr(VI) ions decreased, possibly due to the higher concentration of OH- ions present which competed with Cr(VI) species ($CrO_4^{2-}$) for the adsorption sites. On the other hand, as the adsorption surface (pH > pH zpc) is negatively charged, increasing electrostatic repulsion between a negatively charged Cr(VI) species and a negatively charged adsorbent will also result in a release of the adsorbed ($HCrO_4^-$) and ($CrO_4^{2-}$). Another factor affecting the variation of adsorptive capacity for different levels of pH may be the adsorption free energy of various chromium species ($HCrO_4^-$, $H_2CrO_4$ and $CrO_4^{2-}$) existing at different pH levels (Hu et al., 2005a).

The dramatic decrease of the Cr(VI) uptake with the increase of pH values was mainly due to that higher pH values made the surface of magnetite more negatively charged, which greatly enhanced the electrostatic repulsion between magnetite and Cr(VI) anions, leading to a release of the adsorbed Cr(VI) species from the magnetite surface.

At pH of 2–6, the predominant Cr(VI) species mainly exists in the monovalent $HCrO_4^-$ form, which is then gradually converted to the divalent $CrO_4^{2-}$ form as pH increases. The adsorption free energy of $HCrO_4^-$ is lower than that of $CrO_4^{2-}$, and consequently $HCrO_4^-$ is more favorably adsorbed than $CrO_4^{2-}$ at the same concentration. The removal of Cr(VI) at lower pH is mainly due to the adsorption of $HCrO_4^-$, which is expected to be adsorbed in larger quantities than $CrO_4^{2-}$ under the same adsorption affinity. When $CrO_4^{2-}$ concentration is much higher than $HCrO_4^-$ at higher pH, the adsorption free energy of $CrO_4^{2-}$ is lower, and only under such a circumstance can $CrO_4^{2-}$ adsorption be more favorable than $HCrO_4^-$ (Yuan et al., 2010, Hu et al., 2005a). The surfaces of magnetite nanoparticles in aqueous solution are positively charged and anion adsorption favored at a pH value below the $pH_{ZPC}$ (the zero point of charge, ~8.3) of magnetite, and vice versa, negatively charged and cation adsorption favored when pH is above the $pH_{ZPC}$.

In highly acidic media, the adsorbent surfaces might be highly protonated and favor the uptake of Cr(VI) in the anionic form, $HCrO_4^-$ As a result, an increase of pH value will make the magnetite surfaces less positively charged, greatly weakening the electrostatic attraction between magnetite particles and negatively charged Cr(VI) anions. (Selvi et al., 2001(Yuan et al., 2009).

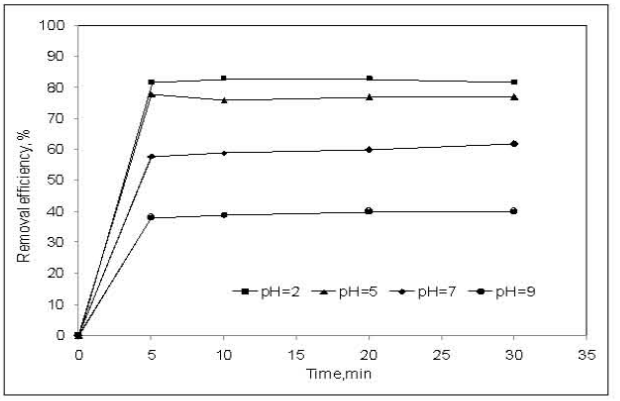


**Fig. 4.** Effect of pH on the removal of Cr(VI) by magnetite nanoparticles

### *3.3. Effect of initial concentration on chromium removal efficiency*

Table 2 indicates that in initial concentrations of 10, 50, 100, and 200 mg Cr/L (pH 2, shaking speed 250 rpm, and nanoparticles doses of 5 g/L) removal efficiency of chromium was reduced from 82 to 18 percent. Therefore, Cr(VI) removal is inversely related inversely to the initial chromium concentrations because the total available adsorption sites are limited, leading to a decrease in percentage of adsorbate removal corresponding to an increased initial adsorbate concentration (Hu et al., 2005a). Also, studies by Niu showed that the chromium removal efficiency decreases with increased initial chromium concentration (Shao-feng et al., 2005).

**Table 2.** Removal efficiency percent of chromium from simulated electroplating industry wastewater, (nanoparticles dose 5 g/L, pH 2, shaking speed 250 rpm)

| ***Cr(VI), mg/L*** | 10 | 50 | 100 | 200 |
|---|---|---|---|---|
| ***Removal efficiency, %*** | 81.9±0.07 | 59±2 | 24±0.6 | 18.05±0.07 |

### *3.4. Effect of common ions on chromium removal efficiency*

The presence of common ions simultaneously with Cr(VI) invariably implies competition for available adsorption sites. Although some adsorption sites can only adsorb certain solutes, the presence of other solutes will, in general, reduce the adsorption of any given solute to some degree. Furthermore, the adsorption of other ions affects the recovery of Cr(VI) directly. Thus, it is necessary to investigate the competitive influence of common anions and cations with Cr(VI). In chrome-plating wastewater, the major cations are $Na^+$, $Cu^{2+}$ and $Ni^{2+}$, and the major anions are $NO_3^-$, $SO_4^{2-}$ and $Cl^-$. As shown in Fig. 5, the influences of $Na^+$ on Cr(VI) were insignificant because they did not compete for the active surface with chromate anions at this pH. The competitive influence of $NO_3^-$ and $Cl^-$ on $CrO_4^{2-}$ adsorption can be very small, but the competition of $SO_4^{2-}$ is significant. Compared with Cr(VI) species, nitrate and chloride are poor ligands and indicate a weaker adsorption mechanism via outer-sphere complexation. The result of this study is consistent with the results of Hu et al. (Hu et al., 2005a). In addition, there was no competitive influence on Cr(VI) from these ions even with further increases in ion concentrations.

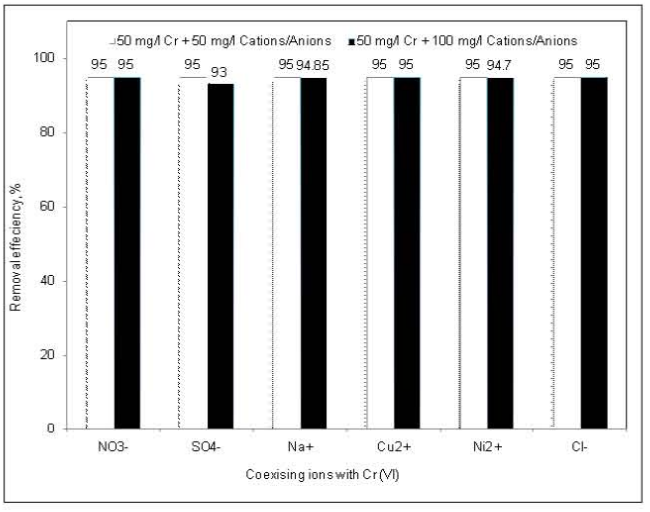


**Fig. 5.** Effect of common ions on the removal of Cr(VI) by magnetite nanoparticles

### *3.5. Effect of shaking speed on chromium removal efficiency*

Removal of chromium at a shaking speeds of 50,100, 200 and 300 rpm were found to be 70.5, 73, 75, and 80%, respectively (pH 2, 0.1 g $Fe_3O_4$ nanoparticles, 100 mg Cr/L solution, Fig. 6). Above 300 rpm chromium removal efficiency was unchanged. The results indicated that the rate of Cr(VI) removal was controlled by the degree of shaking. Increasing the shaking speed decreases the boundary layer and the film resistance to mass transfer surrounding the adsorbent particles (Hu, 2006). Consequently, as shaking speed increases, a reduction in the boundary layer surrounding the particle will occur. Because the system was well-mixed under higher shaking speed, the mass transfer effect became insignificant. On the other hand, the percentage of Cr(VI) removal increased with increasing shaking speed. This can be explained by the fact that for a relatively lower mixing rate the system was incompletely agitated and hence the nanoparticles were poorly dispersed in the solution. This may have resulted in a part of the surface area of the adsorbent being exposed and adsorbing Cr(VI) ions.

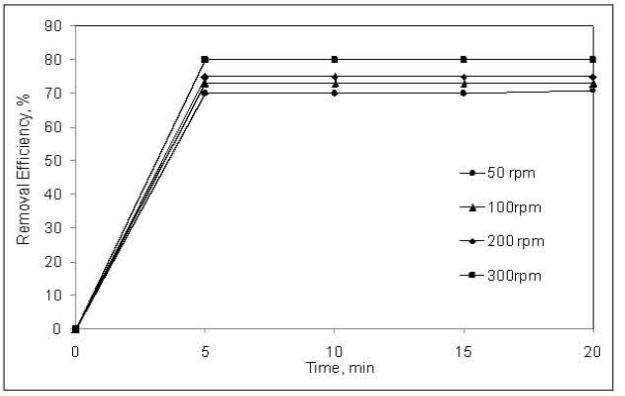


**Fig. 6.** Effect of shaking speed on the removal of Cr(VI) (pH 2, $Fe_3O_4$ nanoparticles dose of 0.1 g, 100 mg Cr/L)

### *3.6. Adsorption isotherms*

In order to model the adsorption behavior and calculate the adsorption capacity, magnetite adsorption isotherms were investigated at different pH levels (2, 5, 7 and 9) and at 20°C by varying the initial concentration of Cr(VI) from 5 to 200 mg/L. Cr adsorption was correlated well with the Freundlich Eq. 2 (Fig. 7):

$$\log q_e = \log K + 1/n \times \log C_e \qquad (2)$$

where $q_e$ is the amount of chromium species adsorbed at equilibrium in mg/g, $C_e$ is the solute equilibrium concentration in mg/L, and $K$ and $1/n$ are Freundlich constants related to adsorption capacity and intensity of adsorption, respectively.

The values of $K$ and $n$ at different pH levels are shown in Table 3. For a given value of initial concentration and pH, the adsorption capacity decreased with increasing in pH, indicating that the lower pH was favored adsorption. In addition, the pH and the Freundlich parameter, log $K$, have a good linear relationship ($R^2$ = 0.98). The dependence of $K$ and $n$ can be explained by surface chemistry in aqueous phases. The surfaces of metal oxides are

usually covered with hydroxyl groups that vary in forms at different pH levels. The removal of Cr(VI) at lower pH is primarily due to the adsorption of $HCrO_4^-$, which is predicted to be adsorbed in larger quantities than $CrO_4^-$ under the same adsorption attraction. When $CrO^{2-}_4$ concentration is much higher than $HCrO_4^-$ concentration, at a higher pH, the adsorption free energy of $CrO_4^{2-}$ is lower. Only under such a situation will $CrO_4^{2-}$ adsorption be better than $HCrO_4^-$ adsorption (Hu et al., 2005a). At low pH, the surface charge is neutral at the point of zero charge (PZC), which is 6.5 for the mesoporous $\gamma$-$Fe_2O_3$. At pH higher than PZC, the cr(VI) adsorption can be explained by anion exchange on the metal-oxide surfaces (Wang and Lo, 2009). In addition the surface charge is neutral at $pH_{pzc}$. Below the $pH_{pzc}$, the adsorbent surface is positively charged, and anion adsorption occurs. With the increase in pH, uptake of Cr(VI) ions decreased, which is apparently due to the higher concentration of $OH^-$ ions present in the reaction mixture, which competed with Cr(VI) species ($CrO_4^{2-}$) for the adsorption sites. On the other hand, as the adsorption surface ($pH>pH_{zpc}$) is negatively charged, increasing electrostatic repulsion between negatively charged Cr(VI) species and negatively charged adsorbent will also result in a release of the adsorbed $HCrO_4^-$ and $CrO_4^{2-}$ (Hu et al., 2005a).

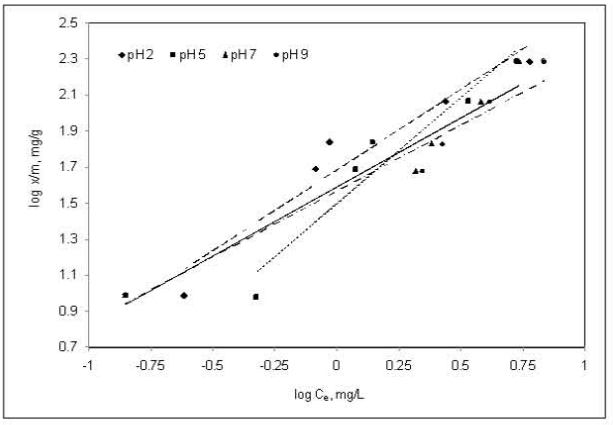


**Fig. 7.** Freundlich isotherms at different pH levels [initial Cr(VI) concentrations: 5, 10, 50, 70, 120, and 200 mg/L; sorbent concentration: 1 g/L; pH 2; agitation rate 250 rpm]

**Table 3.** The Freundlich constants at different pH

| *pH* | *Freundlich constants* | | |
|---|---|---|---|
| | *log K* | *1/n* | $R^2$ |
| 2 | 1.690 | 0.893 | 0.930 |
| 5 | 1.503 | 1.175 | 0.936 |
| 7 | 1.598 | 0.764 | 0.949 |
| 9 | 1.570 | 0.732 | 0.959 |

In this study, the adsorption capacity of the $Fe_3O_4$ for Cr(VI) at pH of 2 was 41 mg/g. This capacity is higher than diatomite (11.55 mg/g), anatase (14.56 mg/g), commercial activated carbon (15.47 mg/g), and 16.13 mg/g for beech sawdust (Wang and Lo, 2009), and also 14.6 mg/g for micron-scaled magnetite (Yuan et al., 2010), 31.5 mg/g for modified Jacobsite ($MnFe_2O_4$), (Hu et al., 2005b), 17 mg/g for siderite (Erdem et al., 2004), 15.3 mg/g for montmorillonite-supported magnetite (Yuan et al., 2009), and was less than the capacity of 69.2 mg/g for diatomite-supported nano-scaled (Yuan et al., 2010).

### *3.7. Chromium removal from real wastewater of electroplating industries*

A sample of chromium wastewater from the electroplating industry with an initial chromium concentration of 56-61 mg/L was used to evaluate chromium removal efficiency for real wastewater. Table 4 shows that the amounts of remained chromium after contacting the electroplating wastewater with magnetite nanoparticles were 10-11 mg/L, indicating an 80% reduction. Thus, organic and suspended material in the wastewater does not significantly affect the removal efficiency of chromium by magnetite nanoparticles.

**Table 4.** Chromium removal efficiency in real wastewater of electroplating industries

| *Chromium concentration( mg/L)* | *Sample name* |
|---|---|
| 59 ± 3 | Raw electroplating wastewater |
| 11 ± 0.8 | After contact with $Fe_3O_4$ |

## 4. Conclusions

Magnetite nanoparticles were applied effectively in the removal of hexavalent chromium from real and simulated electroplating wastewater due to the higher surface area and reactive hydroxyl surface sites. The removal efficiency of Cr(VI) depends on pH, and optimal adsorption occurred at pH 2. The adsorption data was well fitted by the Freundlich isotherm equation. The effects of coexisting ions ($Na^+$, $Cu^{2+}$, $Ni^{2+}$, $SO_4^{2-}$, $NO^{3-}$ and $Cl^-$) on the efficiency of removal were insignificant. The magnetite nanoparticles removed Cr(VI) from real wastewater and were not affected by organic matter. Therefore, it could be concluded that magnetic nanoparticles may be well suited for treating chromium wastewater. However, further research should attempt to improve the adsorption capacity of magnetic particles and apply this method to the removal of metallic elements in large-scale.

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